LA-UR-21-28515
Approved for public release; distribution is unlimited.

Title: Production Facility Prototype Blower 1000 Hour Test Results V

Author(s): Wass, Alexander Joseph
Dogruel, David
Woloshun, Keith Albert

Intended for: Report

Issued: 2021-08-26

Production Facility Prototype Blower 1000 Hour Test Results V

8/16/2021

Alexander Wass, David Dogruel, Keith Woloshun

Introduction

Long duration operations tests of the Aerzen GM 12.4 roots style blower in a closed loop configuration provides valuable data and lessons learned for long-term operation at the Mo-99 production facility. The blower was operated in a closed loop configuration with the flow conditions anticipated in plant operation with a Mo-100 target inline. The additional thermal energy generated from beam heating of the Mo-100 disks was not included in these tests. Thirteen long duration 500 to 1000 hour tests have been completed since the first test was performed in January of 2016. All thirteen long duration tests have proven successful in exposing preventable issues related to oil and helium leaks. All blower tests to this date have resulted in stable blower performance and consistency. A summary of the results for the end of the thirteenth test is included in this report. Complete details for the previous tests are shown in the reports, Production Facility Prototype Blower 1000 Hour Test Results[1], II[2], III[3], and IV[4].

Thirteenth Long Duration Operations Test – 1000 Hours

The pressure vessel was closed and torqued to 1300 lb-ft using the BLUE-GARD gasket in preparation for the thirteenth long duration test. The test was performed with helium for 1000 hours between 9/1/20 to 2/16/21. The test section gate valve was used to achieve higher flow rates closer matching the production target design. The pressure vessel on the production prototype helium flow system was pressurized with helium to 2068 kPa (300 psig). The blower speed was set to 1800 RPM to achieve a high pressure drop that was below the torque limits of the blower. The blower torque was about 93 N-m, and the helium mass flow rate was 283 g/s with a differential pressure of about 102 kPa (15 psi). The rise in gas temperature through the blower was about 9°C.

From the start of the long duration test, the helium leak rate was abnormally high (approximately one helium cylinder per day). Therefore, after the first two weeks of operation, the blower was turned off to diagnose the leak. There were two causes of the leak. The first was the pressure relief valve (PRV) that had a slow leak. The PRV leak was fixed by pushing and pulling on the test ring, which re-seated the valve. The other cause of the leak was due to the pressure vessel fasteners and gasket. The gasket was cut undersized by the vendor, which did not create an acceptable seal, and the fasteners had galled over time during the more than thirteen installation cycles. The galled fasteners made it difficult to install and thus, proper torque values were questionable. Therefore, both the gasket and fasteners were replaced with new components. This includes all nuts, bolts, and washers. Figure 1 shows the galled fasteners compared to the new fasteners. Once closed and filled with helium, the leak rate was as expected.

[1] K. Woloshun, LA-UR-16-27971, Production Facility Prototype Blower 1000 Hour Test Results, 2016.
[2] A. Wass, LA-UR-18-20146, Production Facility Prototype Blower 1000 Hour Test Results II, 2018.
[3] A. Wass, LA-UR-18-29897, Production Facility Prototype Blower 1000 Hour Test Results III, 2018.
[4] A. Wass et al., LA-UR-19-31253, Production Facility Prototype Blower 1000 Hour Test Results IV, 2019.

Figure 1 – Old vs. new fasteners

In order to visualize any oil leaks that may occur during the test, a high-pressure rated camera was installed in the pressure vessel to display and record real-time video of the blower during operation. A DEEPSEA Power & Light LED Multi SeaCam model MSC-2065 camera was used. The pressure rating on this camera is 5600 psig (water) and is more than suitable for the conditions within the pressure vessel. The camera recorded video during the entire long duration test and did not show any signs of oil leaks. Images at the beginning and end of the test can be seen in Figs. 2-3. Note that the bottom images are blurry due to electrical noise from the motor during operation. The noise disappeared once the motor was turned off during weekly five minute restarts.

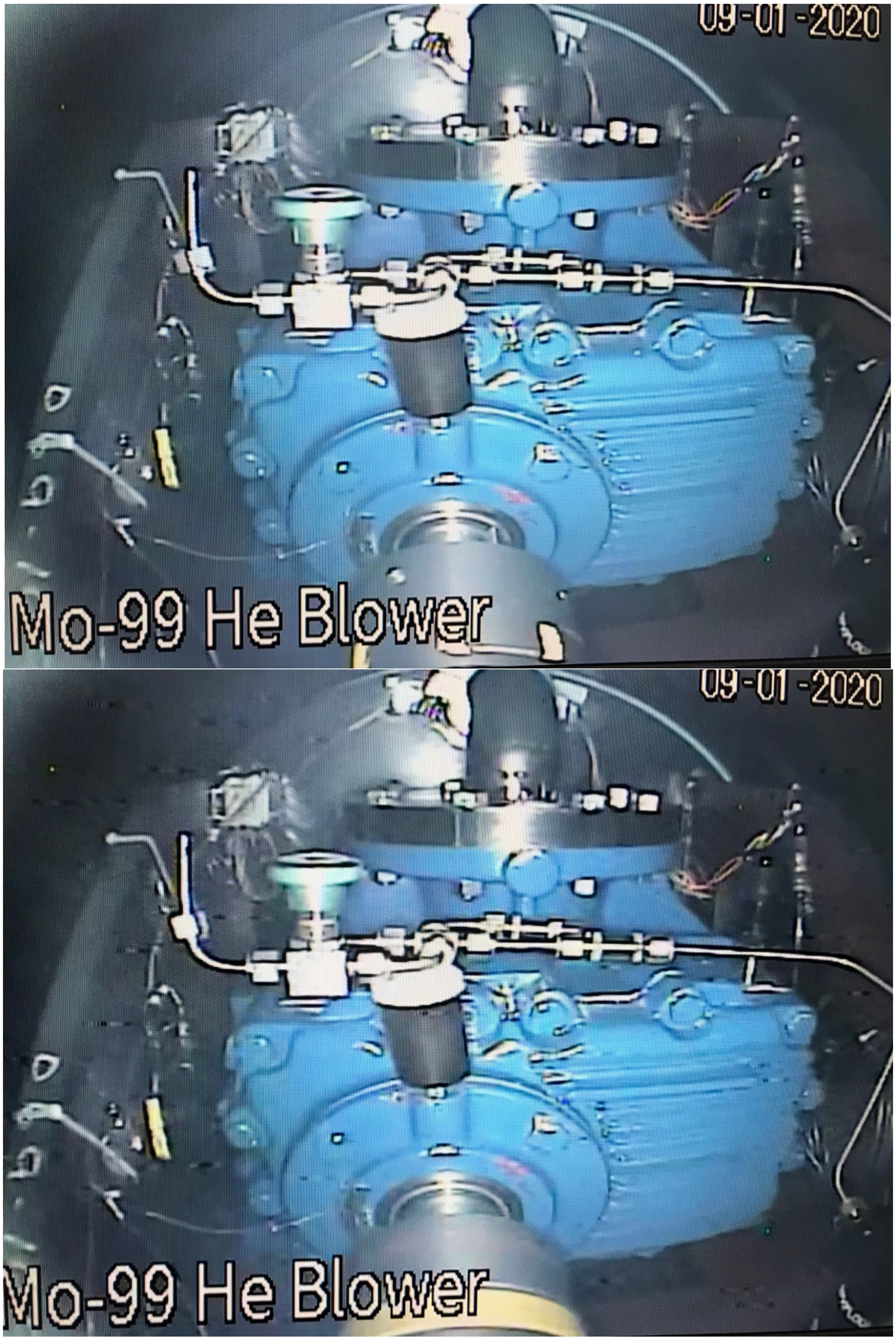


Figure 2 - High pressure camera image at the start of the 13th test with motor off (top) and on (bottom)

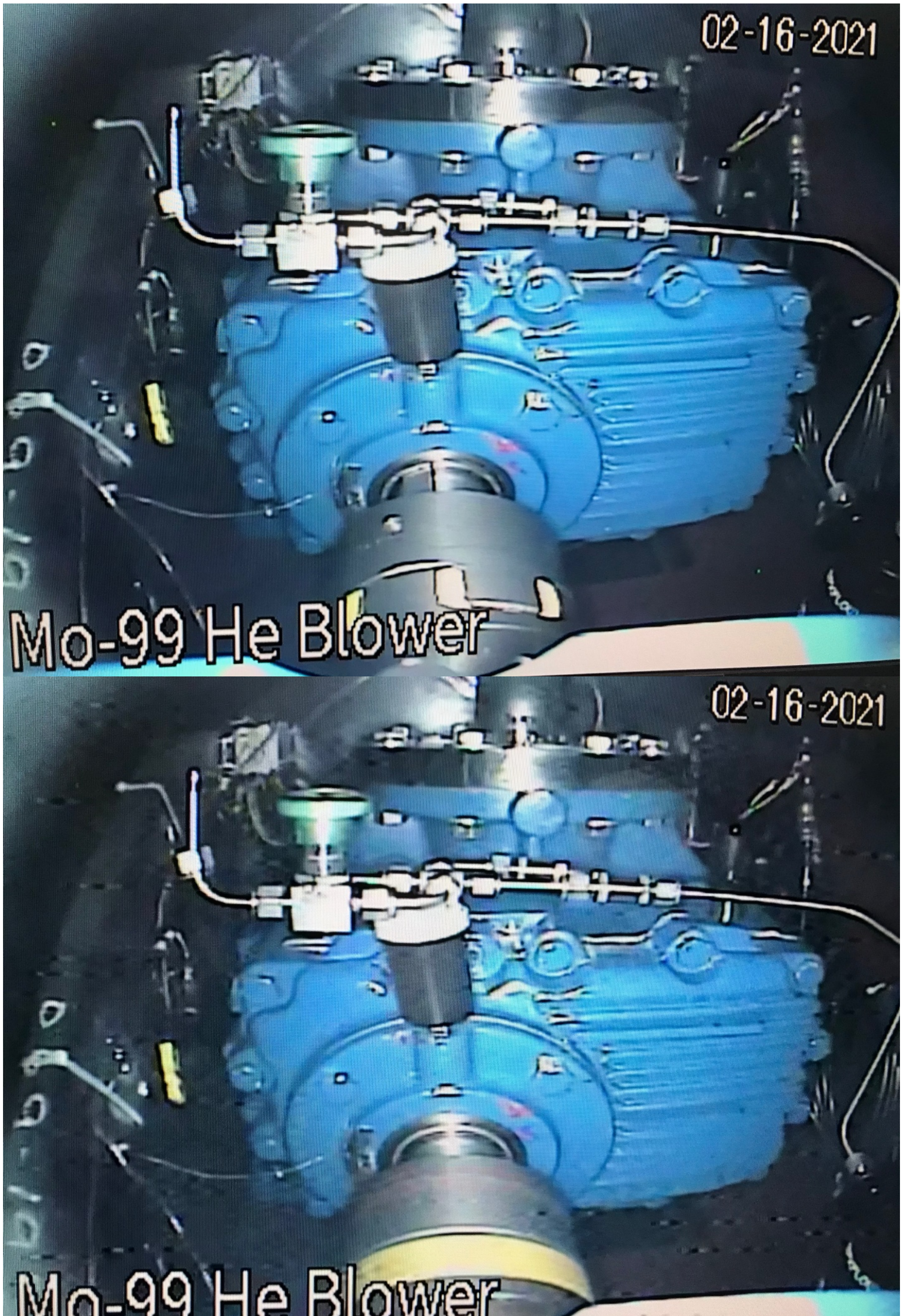


Figure 3 - High pressure camera image at the end of the 13th test with motor off (top) and on (bottom)

During the long duration test, a Residual Gas Analyzer (RGA) was used to sample the helium for hydrocarbons during operation. An in-depth explanation of the RGA system can be read in the report, Final Report Helium Sample Loop Monitoring System[5], by William Hollis and David Dogruel. The goal for this long duration test was to determine the presence of oil in the helium if there was an oil leak from the blower. Once per week, the RGA 300 was scanned multiples times. Each scan consisted of a 40 scan average between 2-100 and 2-299 AMU at a scan speed of 5. First, the RGA was scanned at vacuum to record baseline values typically in the mid to high 10^-8 Torr range. Next, helium from the pressure vessel was introduced slowly into the RGA via a variable leak valve until the vacuum pressure increased to ~1.8E-5 Torr. The RGA did not detect any hydrocarbons within the helium during the duration of this test, however, it is difficult to determine if oil is actually present in the helium or is not detectable by the RGA. A summary of the RGA results is described in greater detail at the end of this report.

Upon completion of this test, the pressure vessel was depressurized and opened to determine if the internal camera and RGA results were consistent. No oil leaks were found during this test. The blower surfaces can be seen in Fig. 4. Figure 5 shows the oil levels at the rear and shaft-side gear boxes. The shaft-side gear box was dark due to grease leaking into the oil. At the beginning of this test, grease was pumped into the shaft side bearing to make up for losses during the previous test, however, too much grease was added and leaked into the oil, causing the discoloration.

Figure 4 – Oil-free blower surface after thirteenth test

[5] W. Hollis and D. Dogruel, LA-UR-18-25555, Final Report Helium Sample Loop Monitoring System, 2018

Figure 5 – Non-shaft side gearbox oil level (left) and shaft side oil level (right)

Conclusion

The end of the thirteenth long duration test proved successful in showing that the blower is leak tight. The internal high pressure camera was successful in providing real time video of the blower during operation and confirmed that there were no oil leaks. Although further studies need to be performed to determine the effectiveness of RGA scans, they did not detect oil in the helium. Overall, these were very successful long-duration tests, and important operational items continue to be learned.

Summary of review of RGA mass spectral data from Northstar He loop

D. Dogruel, C-CDE

05/17/2021

Introduction

From the Safety Data Sheet (SDS) for pdblowers Roots Synthetic Lubricant ISO VG at https://cdn.pdblowers.com/wp-content/uploads/2016/11/howden_roots_sds__vg_150.pdf two components of this proprietary lubricant formulation can be determined and are listed in Table 1, verbatim from the SDS, below. These two components comprise the only "knowns" in this lubricant and subsequent examination of the mass spectral data focused on the identification of any ion signals from either of these compounds.

CAS number/other identifiers

CAS number : Not applicable.

Ingredient name	**%**	**CAS number**
Butene, homopolymer (products derived from either/or But-1-ene/But-2-ene)	≥10 - <25	9003-29-6
1,2-Benzenedicarboxylic acid, di-C9-11-branched alkyl esters, C10-rich	≥3 - <5	68515-49-1

Any concentration shown as a range is to protect confidentiality or is due to batch variation.

There are no additional ingredients present which, within the current knowledge of the supplier and in the concentrations applicable, are classified as hazardous to health or the environment and hence require reporting in this section.

Table 1: Ingredients in pdblowers Roots Synthetic Lubricant ISO VG from product SDS.

The first, a butene homopolymer (polybutenes) derived from either But-1-ene or But-2-ene, has repeat units as depicted in Figure. 1. The chemical formula of one X and one Y unit is C8H16 with a nominal molecular weight of 112 amu. The degree of polymerization, and thus the molecular weight distribution (M_w) cannot be determined based on the SDS information. A cursory literature review did not reveal vapor pressure values for this polymer at elevated temperatures as would be encountered in the Northstar helium loop blower, thus it is undetermined, based on unknown vapor pressure, whether or not there may be a detectable amount of this compound in the volumes of gas sampled and analyzed by RGA mass spectrometry.

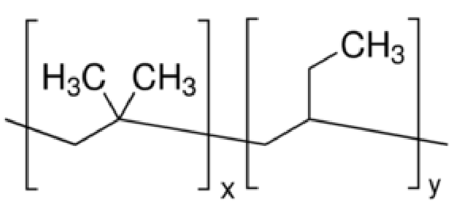
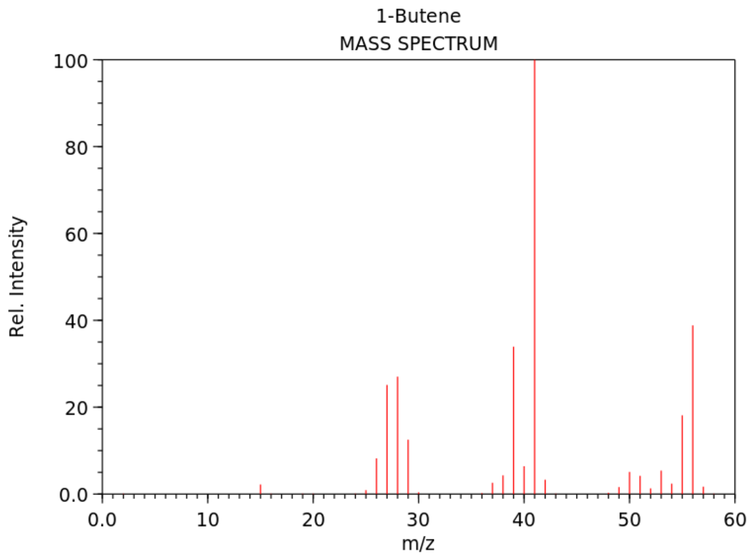
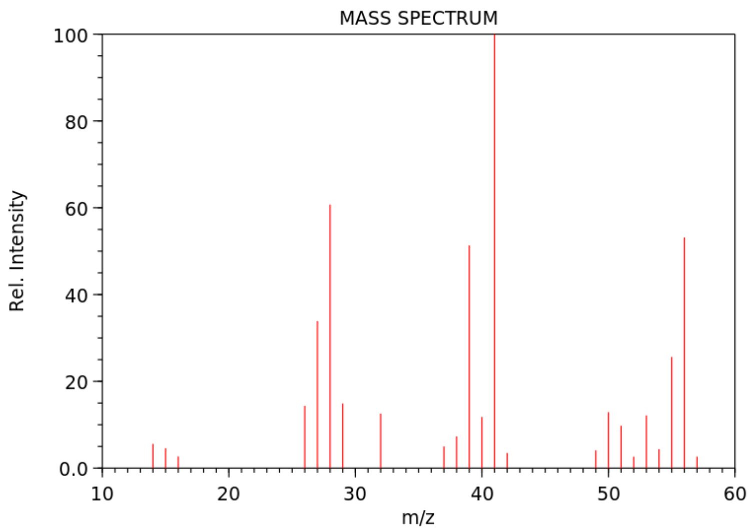


Figure 1: Structure of subunits of polybutene

The structural subunits, and their functional groups, of the polymer can be used to generally predict mass spectral fragmentation patterns. For example, if some amount of the polymer was thermally degraded or otherwise fragmented, a subunit consisting of But-1-ene (1-Butene), C_4H_8 if present in the gas phase upon sampling and analysis by the RGA mass spectrometer, would produce the first mass spectrum as shown in Figure 2. Similarly, a subunit of But-2-ene (2-Butene), C_4H_8 would produce the second mass spectrum of Figure 2.



NIST Chemistry WebBook (https://webbook.nist.gov/chemistry)



NIST Chemistry WebBook (https://webbook.nist.gov/chemistry)

Figure 2: Electron impact (EI) mass spectra of 1-Butene (upper) and 2-Butene (lower)

The diagnostic fragment ions for either/both of these compounds are m/z 56, 55, 41 and 39. The fragment ions at m/z 26, 27, 28 and 29 have potential interferences from the N_2 peak at m/z 28.

The second known constituent of the oil, 1,2 Benzenedicarboxylic acid, di-C9-11-branched alkyl esters C10 rich, also called didecyl phthalate, has a nominal molecular weight of 446.7 amu and is present at only a low concentration (approximately 4%). The structure of this compound, with a chemical formula of C28H46O4 is shown in Figure 3.

The mass spectrum of this compound and its fragment ions is shown in Figure 5. The diagnostic ion for this molecule is m/z 149. The parent ion at m/z 307 is beyond the mass range (300 amu) of the SRS RGA and is not detectable with this instrument.

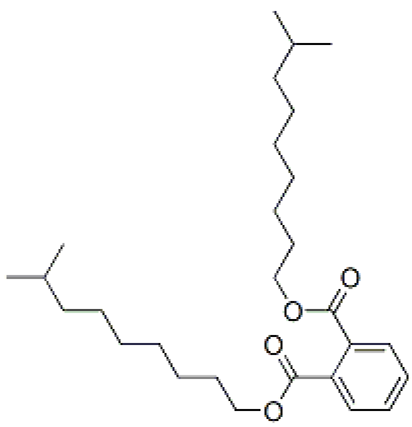


Figure 4: Structure of 1,2 Benzenedicarboxylic acid, di-C9-11-branched alkyl esters C10 rich (didecyl phthalate).

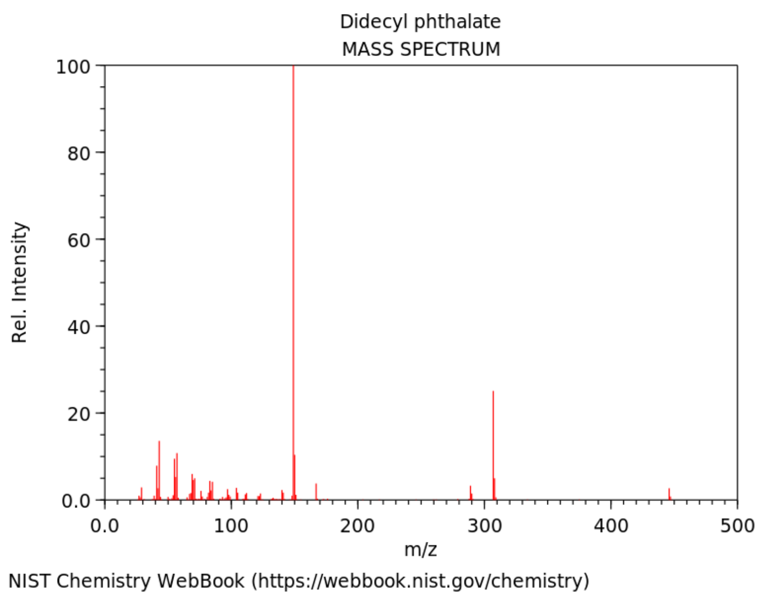


Figure 5: Electron impact (EI) mass spectrum of 1,2 Benzenedicarboxylic acid, di-C9-11-branched alkyl esters C10 rich, also called didecyl phthalate.

Analysis of mass spectra

Prior to examining the mass spectra for presence of the diagnostic ions of the two known constituents, above, the nominal mass accuracy of the RGA was checked using the low mass (2-100 amnu) portion of the mass spectrum from the sample RGA Baseline Helium 1-25-21 (Figure 6). All of the significant signals can be attributed to ions of known molecules as shown in the reference spectrum of ambient air shown in Figure 7, with the masses of all peaks recorded at the nominally correct m/z. Over this mass range (2 to 100 amu), the mass accuracy of the RGA is acceptable.

With the mass accuracy of the RGA, at least up to 100 amu, established, the .txt data files from all samples were plotted (below). Some of the spectra had regions of ion signal that were recorded as negative values for unknown reasons (not plotted here). The data were processed by taking the absolute value of the ion signals at each recorded mass and plotting these positive values on the y axes.

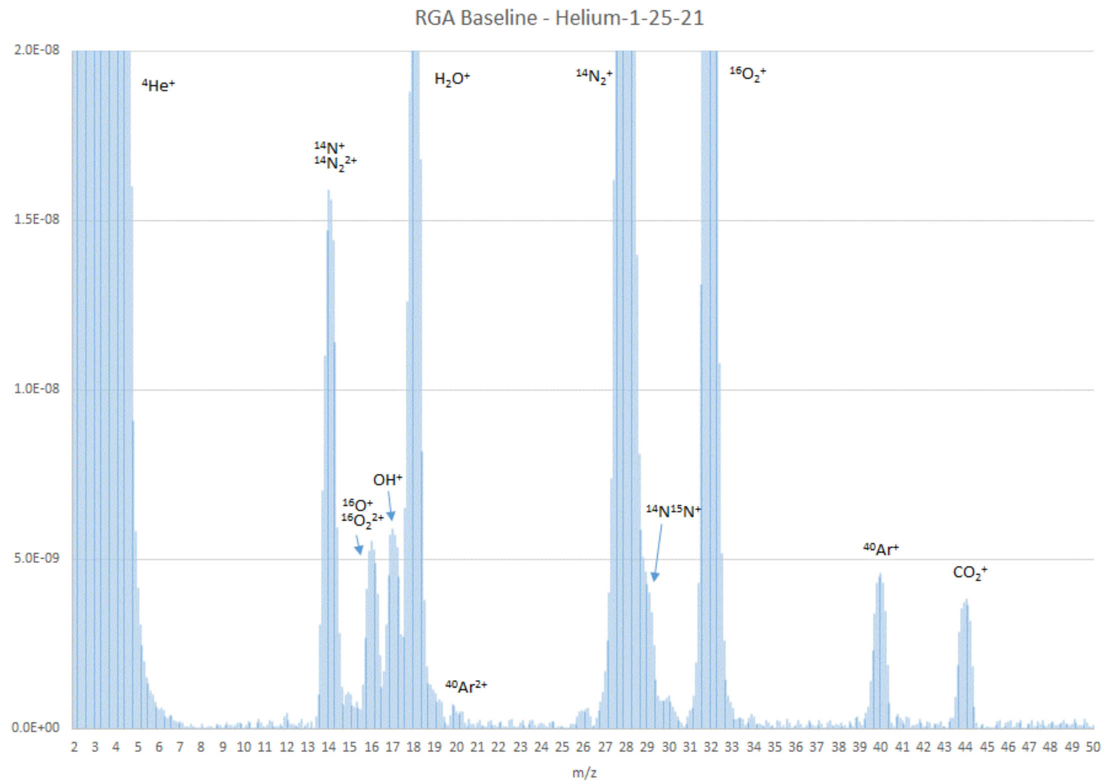


Figure 6: Low mass region of mass spectrum of sample RGA Baseline Helium 1-25-21.

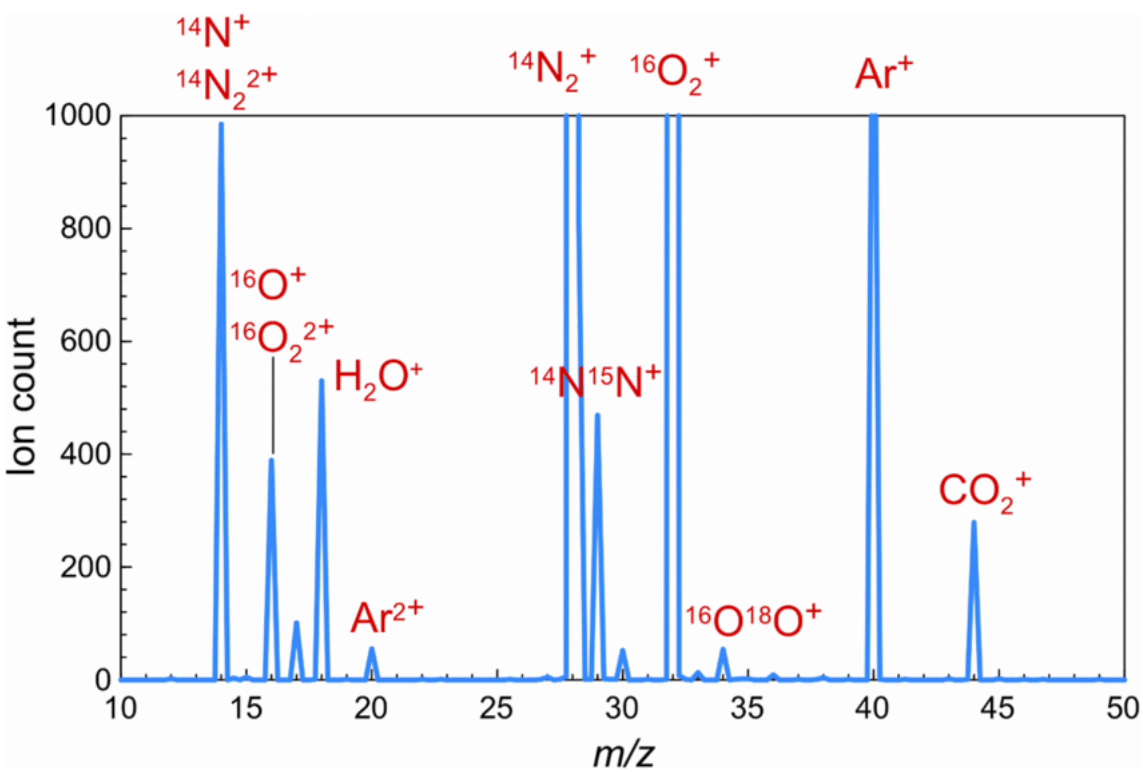


Figure 7: Reference electron impact ionization (EI) mass spectrum of ambient air with vertical axis scale adjusted to show low abundance ion signals.

The y-axis scale of each plot was adjusted to amplify the appearance of any ion signals that are not readily attributable to those observed in the background spectra, similar to the low mass region of the sample RGA Baseline Helium 1-25-21. Each spectrum of a sample other than those identified as baseline was examined for the presence of any of the diagnostic ion signals at m/z 56, 55, 41 and 39 for polybutene and m/z 149 for didecyl phthalate.

The sample spectra were also examined for a common indicator of hydrocarbons, the presence of two or more peaks that are spaced 14 amu (-CH2-) apart.

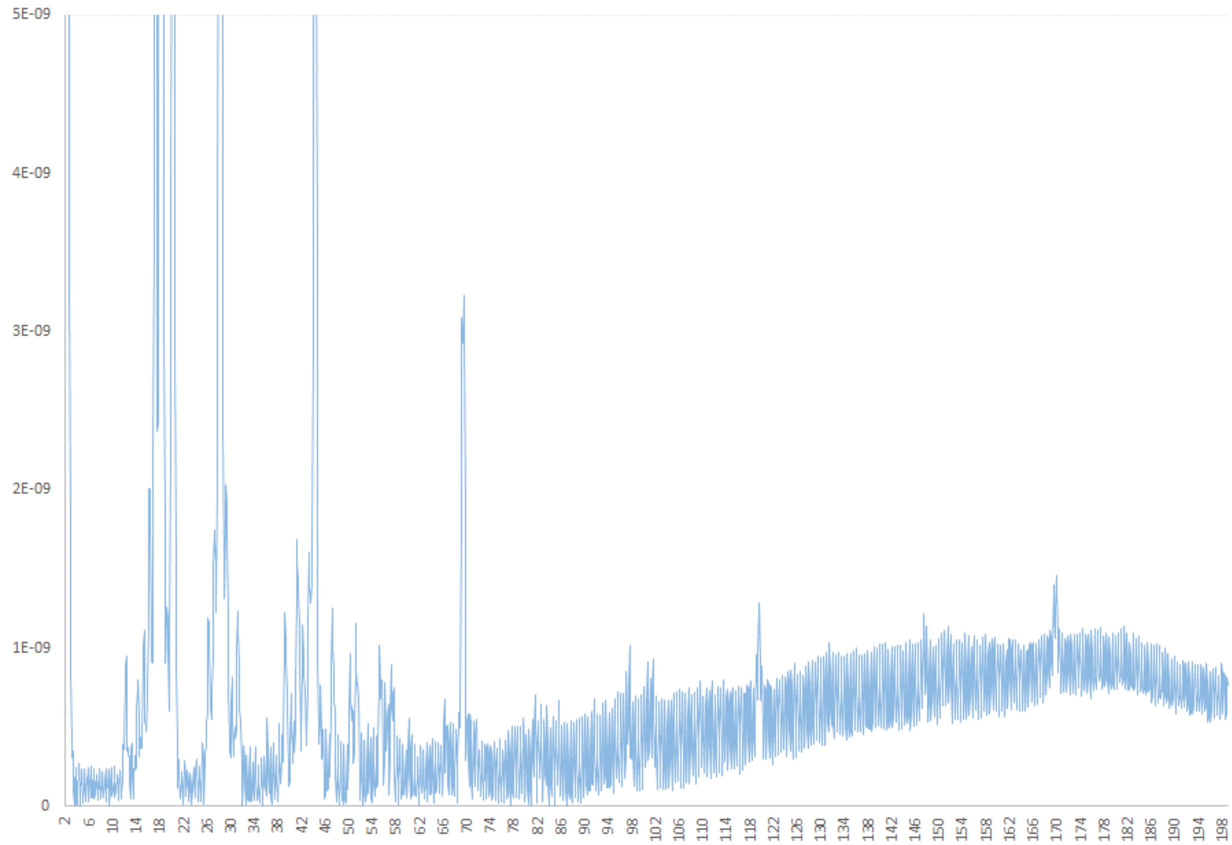
Results

None of the sample mass spectra of samples of helium from the loop contained ion signals indicative of the diagnostic ions for the two known constituents of the oil. As mentioned previously, it is possible that (1) no oil is present in the helium loop, (2) no constituent of the oil is present in the helium at a concentration that is detectable with the sampling system and RGA and/or (3) no constituent of the oil, if present in the helium at a potentially detectable concentration, has a vapor pressure such that a detectable amount of any constituent is able to be sampled and analyzed by the RGA.

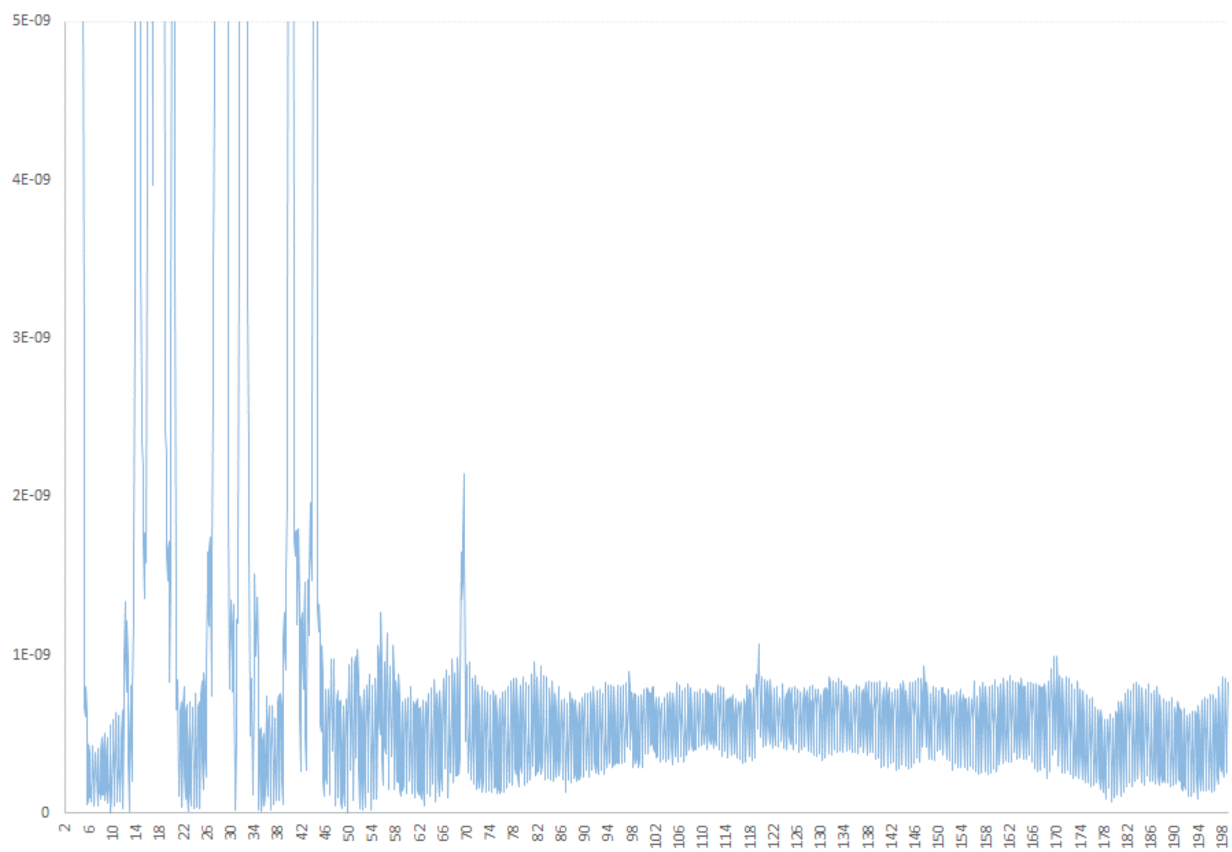
None of the sample mass spectra contained resolved peaks spaced 14 amu apart, suggesting that no hydrocarbons due to the blower oil were detected by the sampling and RGA system (considering the possibilities 1 through 3, above).

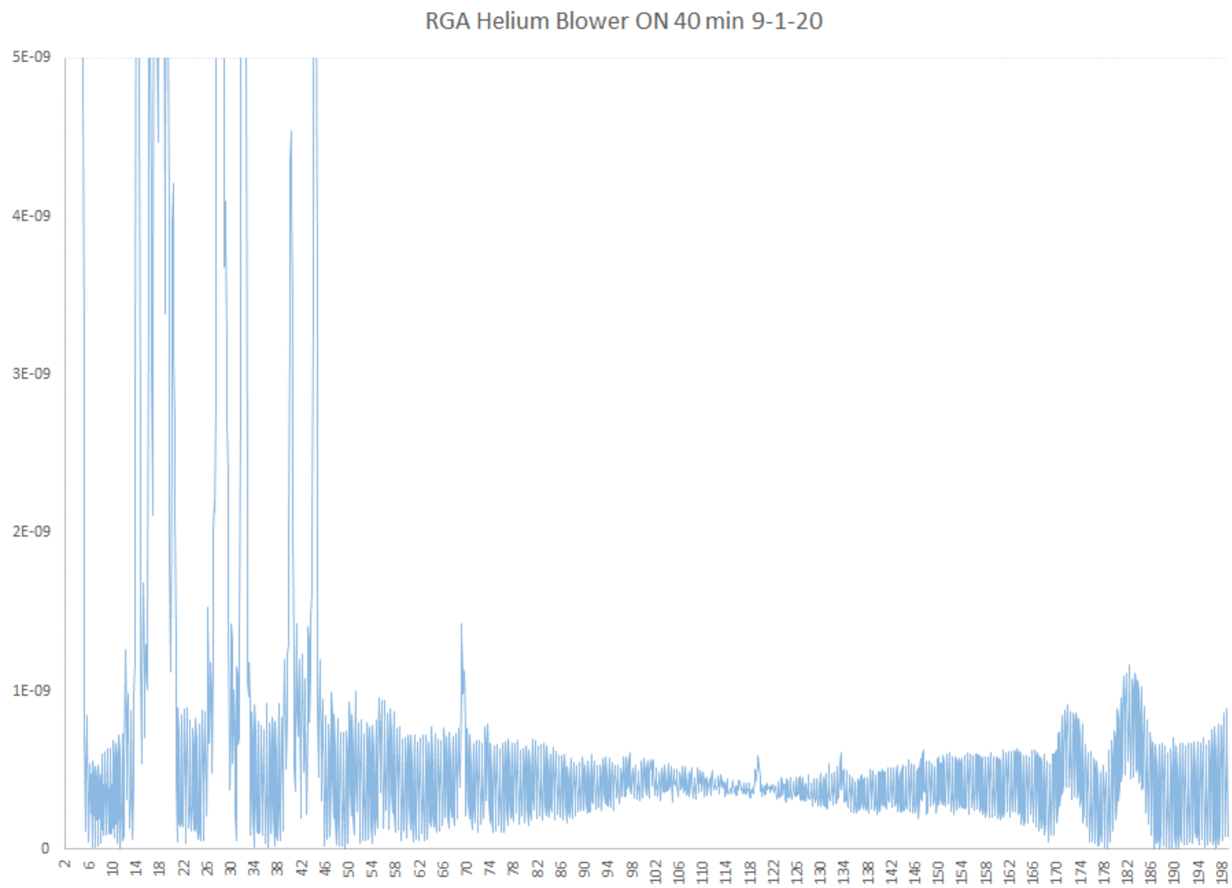
As stated in a previous report on the assembly and testing of the RGA sampling and analysis system for the Northstar helium loop, the analysis of samples of the helium gas with complementary spectroscopic techniques that have the ability to indicate the presence of polymeric compounds and other compounds at masses above 300 amu may be considered.

RGA Baseline - Vacuum 9-1-20

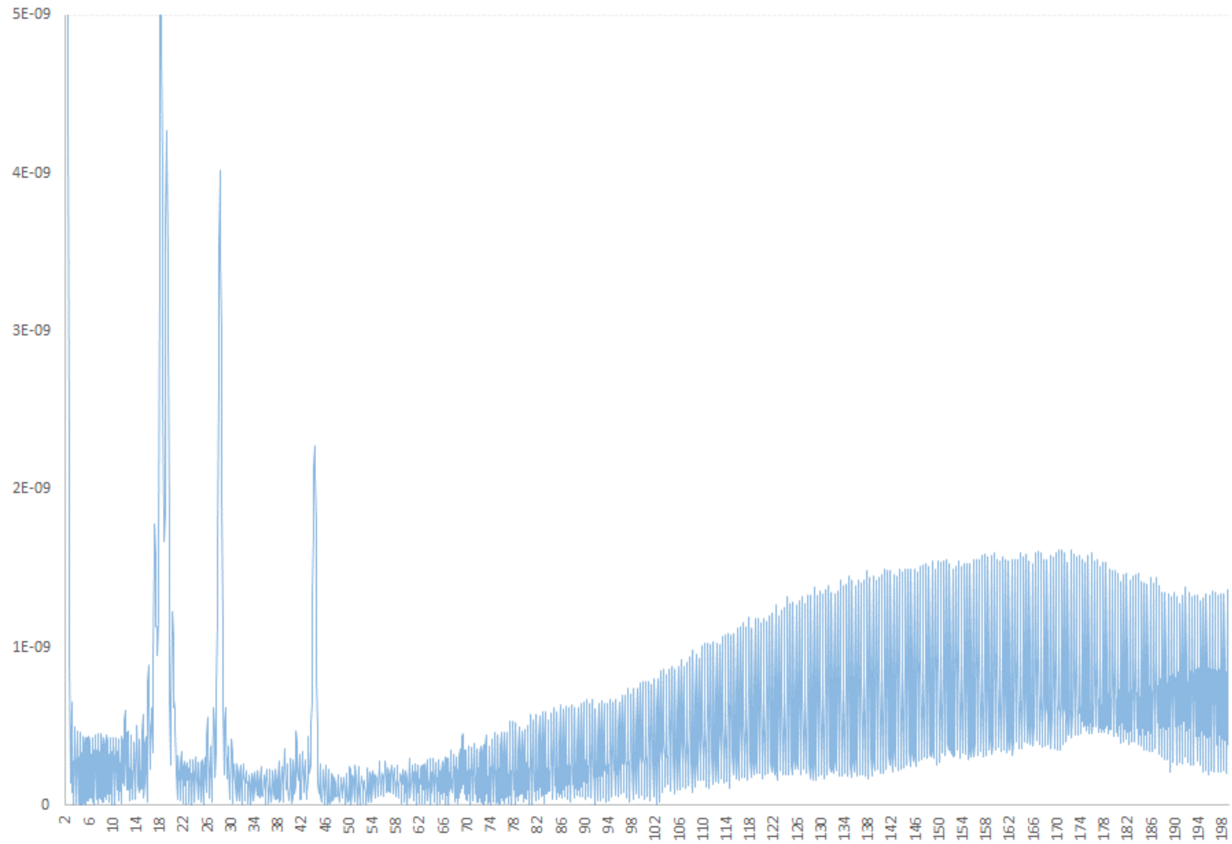


RGA Baseline - Helium 9-1-20

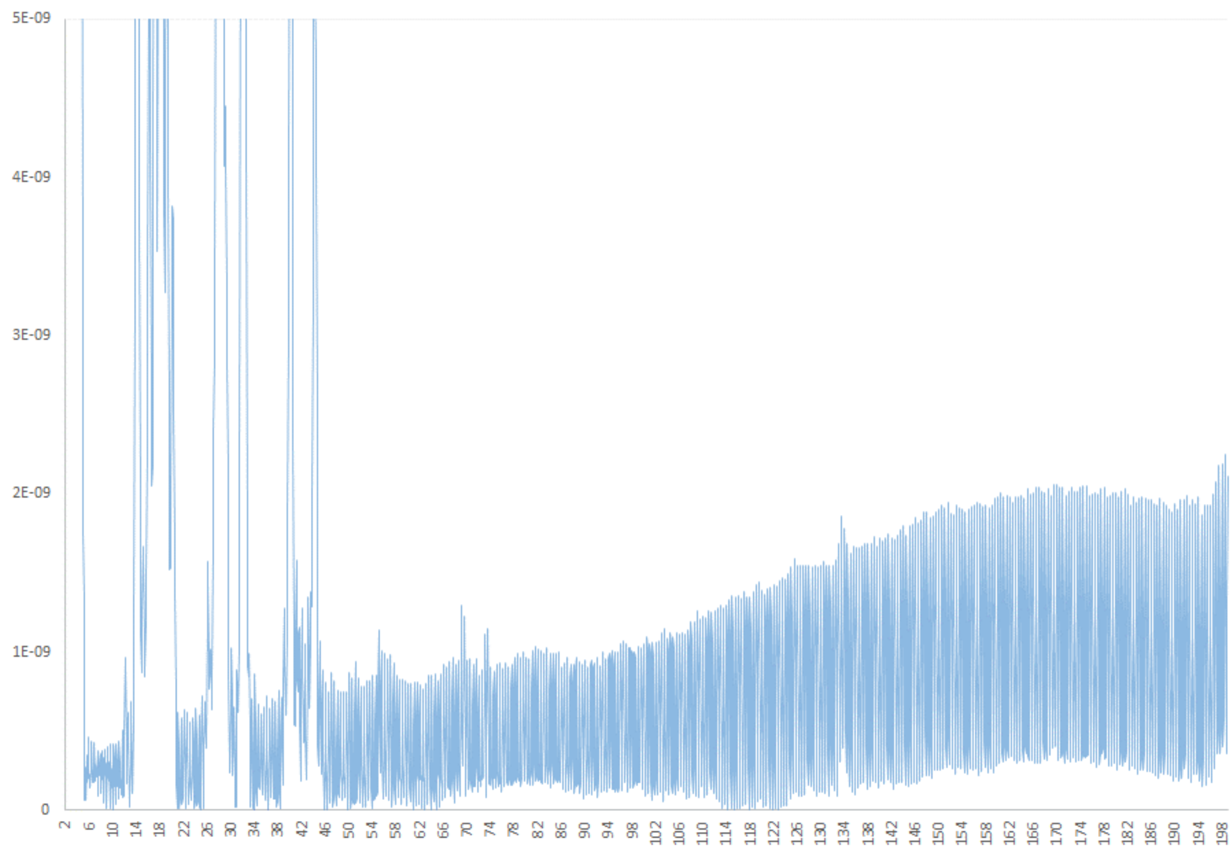


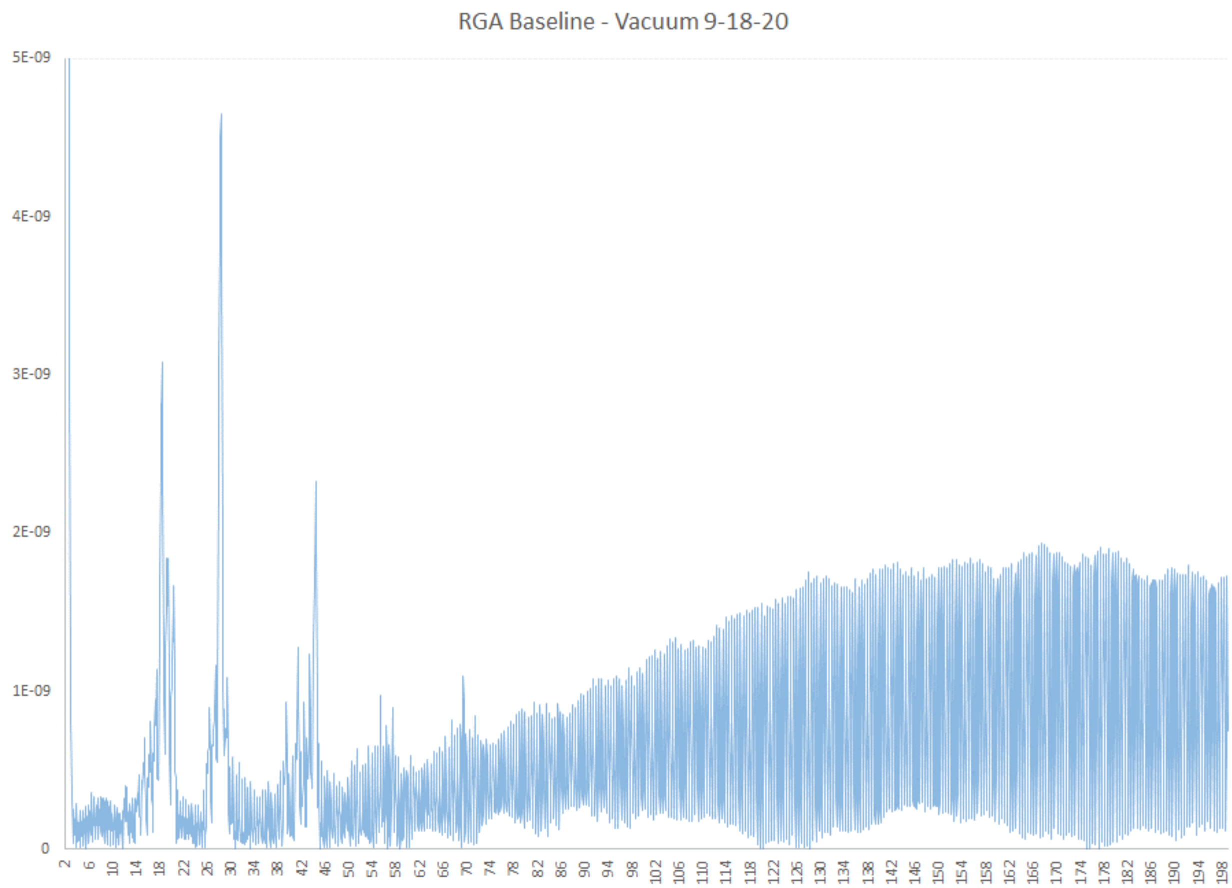

RGA Helium Blower ON 40 min 9-1-20
5E-09
4E-09
3E-09
2E-09
1E-09
0
2
6
10
14
18
22
26
30
34
38
42
46
50
54
58
62
66
70
74
78
82
86
90
94
98
102
106
110
114
118
122
126
130
134
138
142
146
150
154
158
162
166
170
174
178
182
186
190
194
198

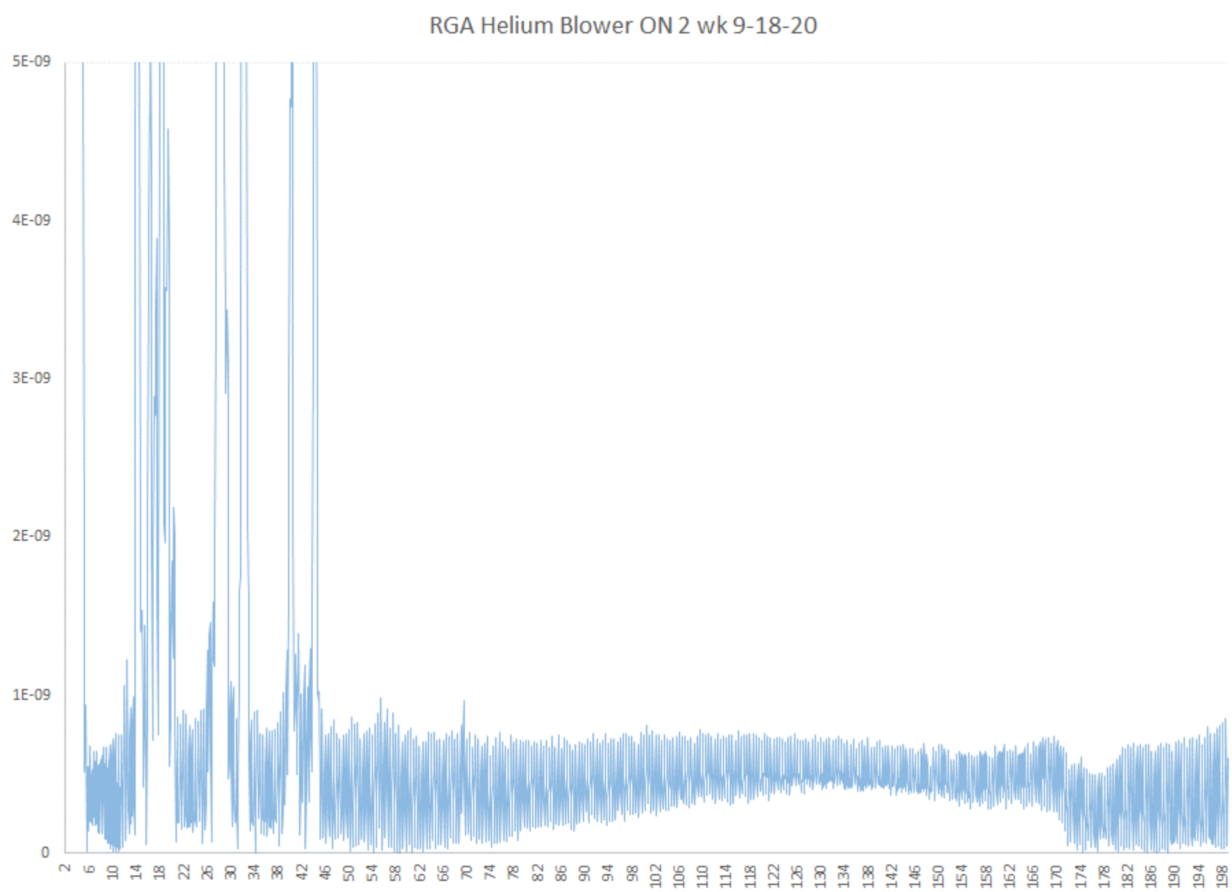
RGA Baseline - Vacuum 9-9-20



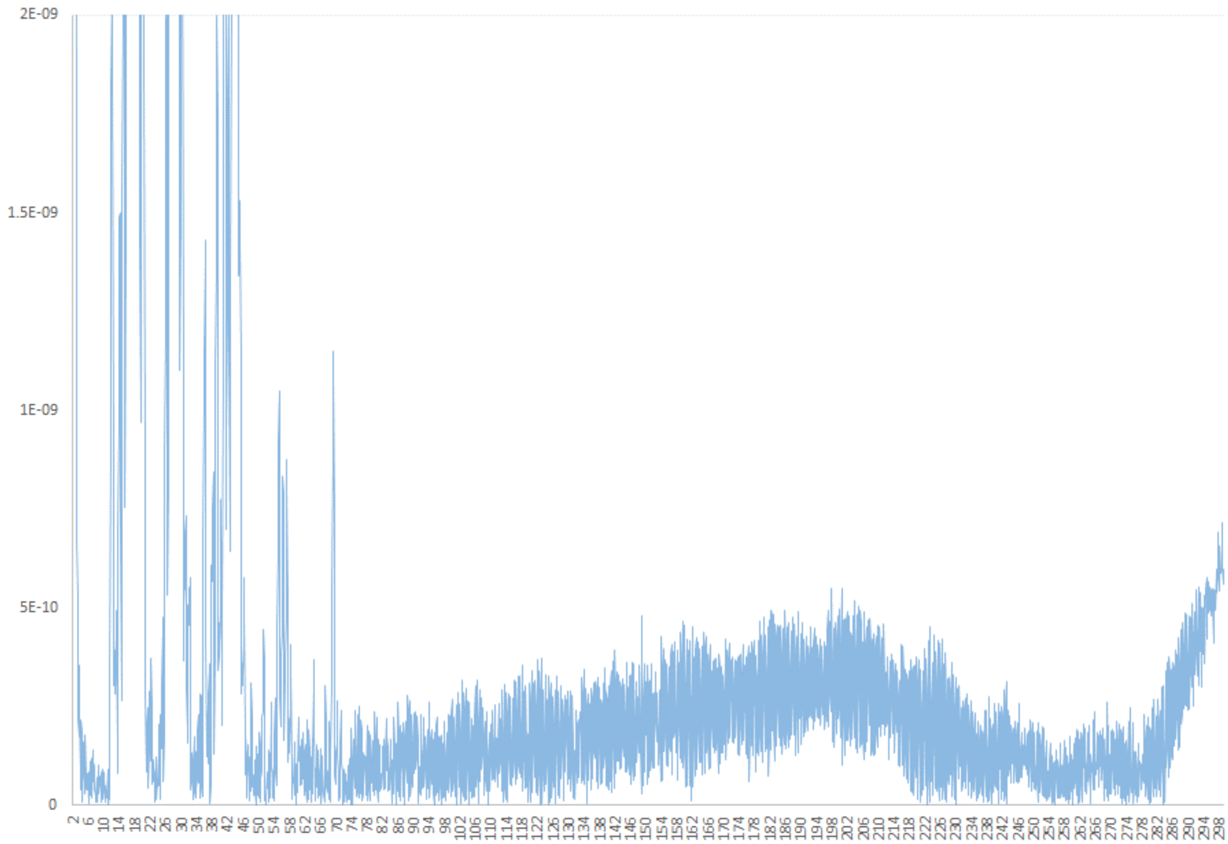
RGA Helium Blower ON 1 wk 9-9-20



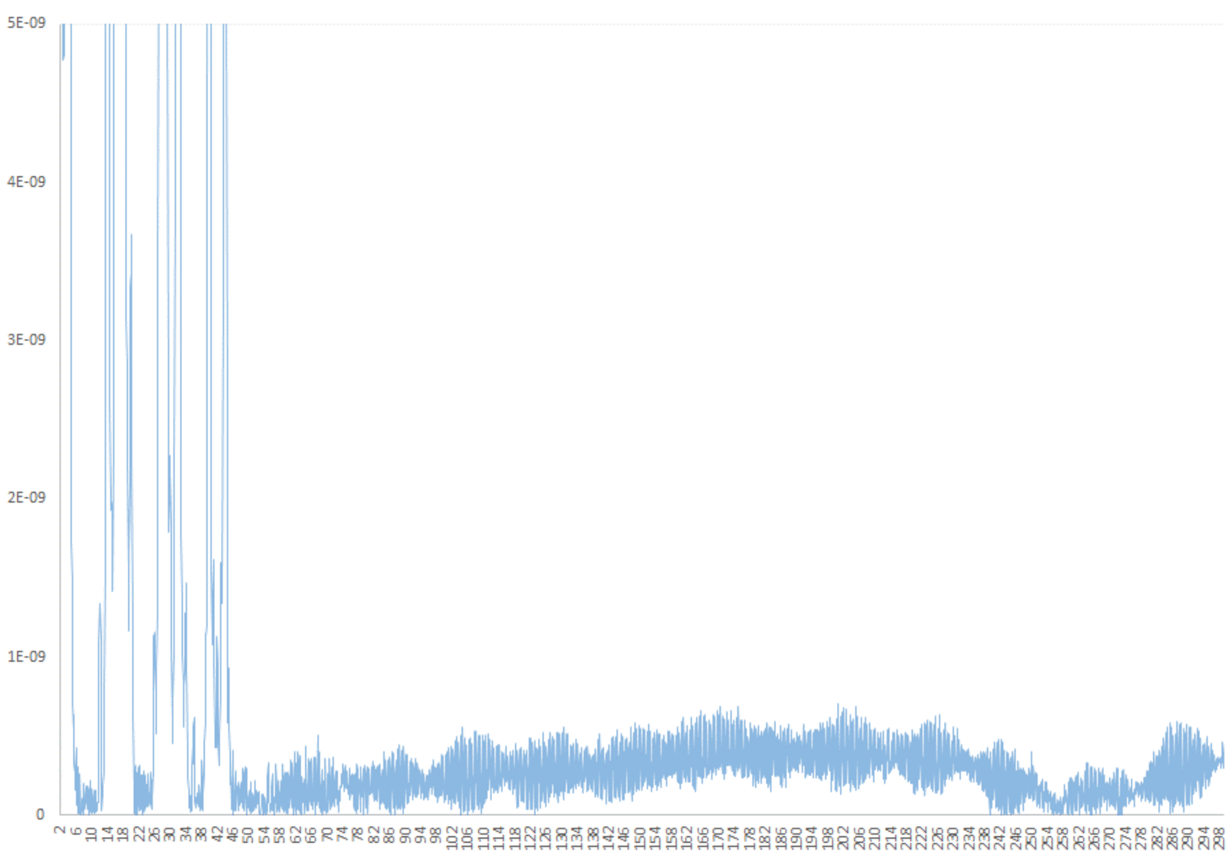

RGA Baseline - Vacuum 9-18-20
5E-09
4E-09
3E-09
2E-09
1E-09
0

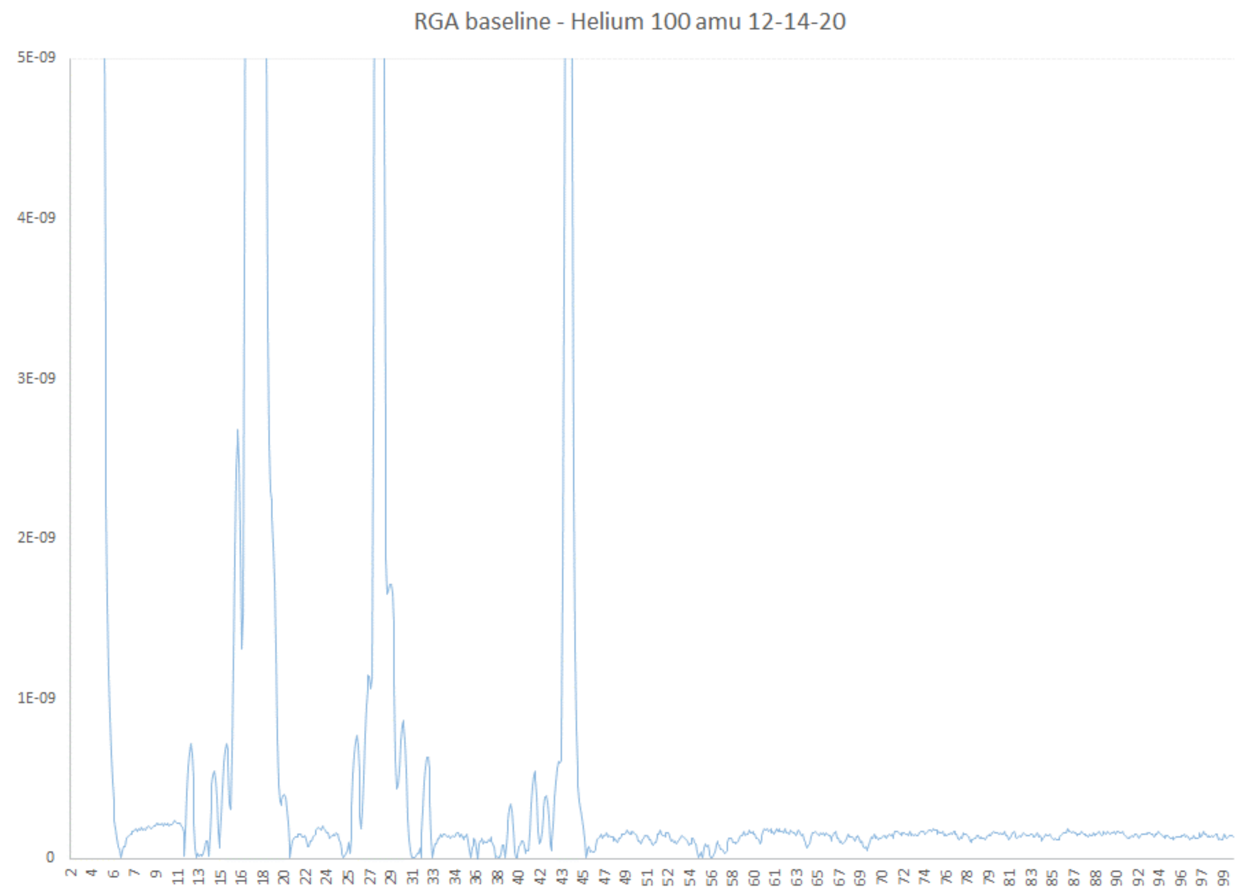

RGA Helium Blower ON 2 wk 9-18-20
5E-09
4E-09
3E-09
2E-09
1E-09
0

RGA Baseline - Vacuum 12-14-20

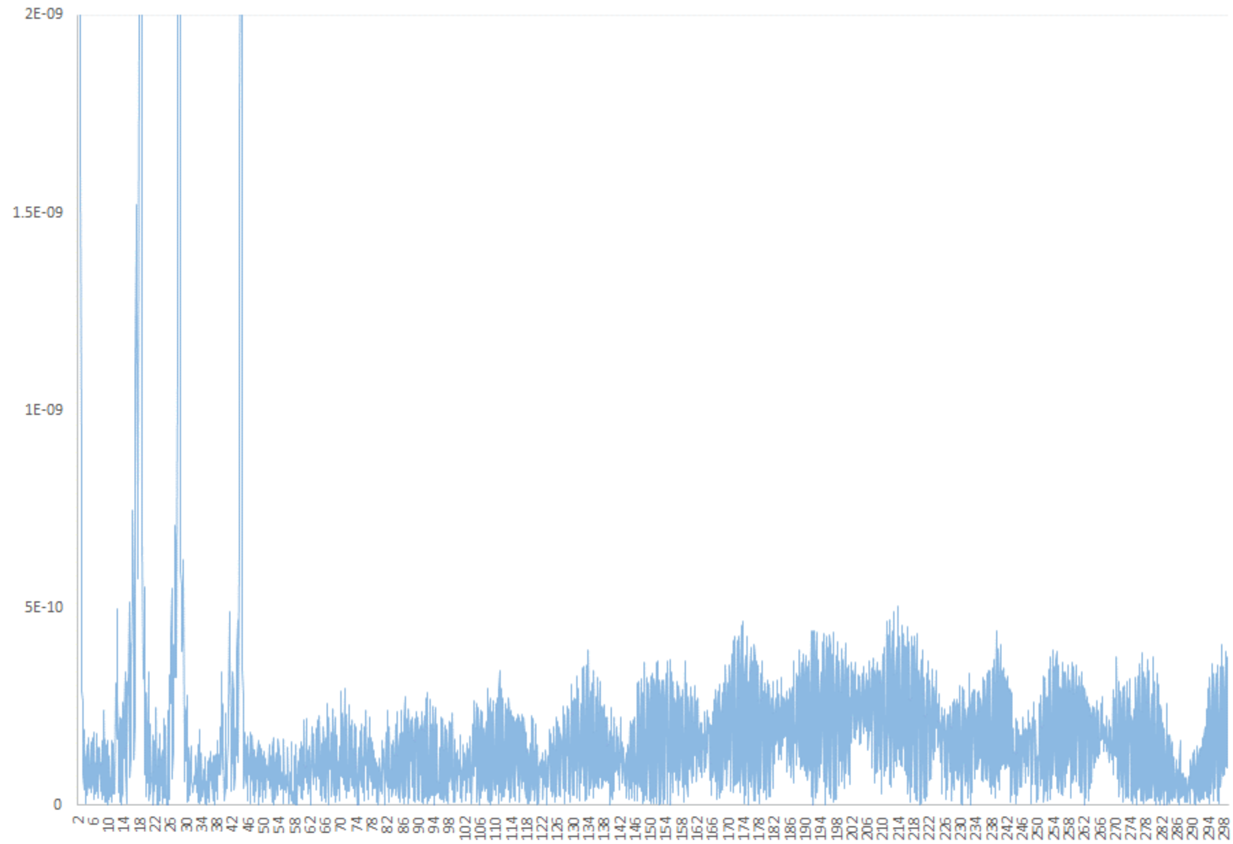


RGA Baseline - Helium 12-14-20

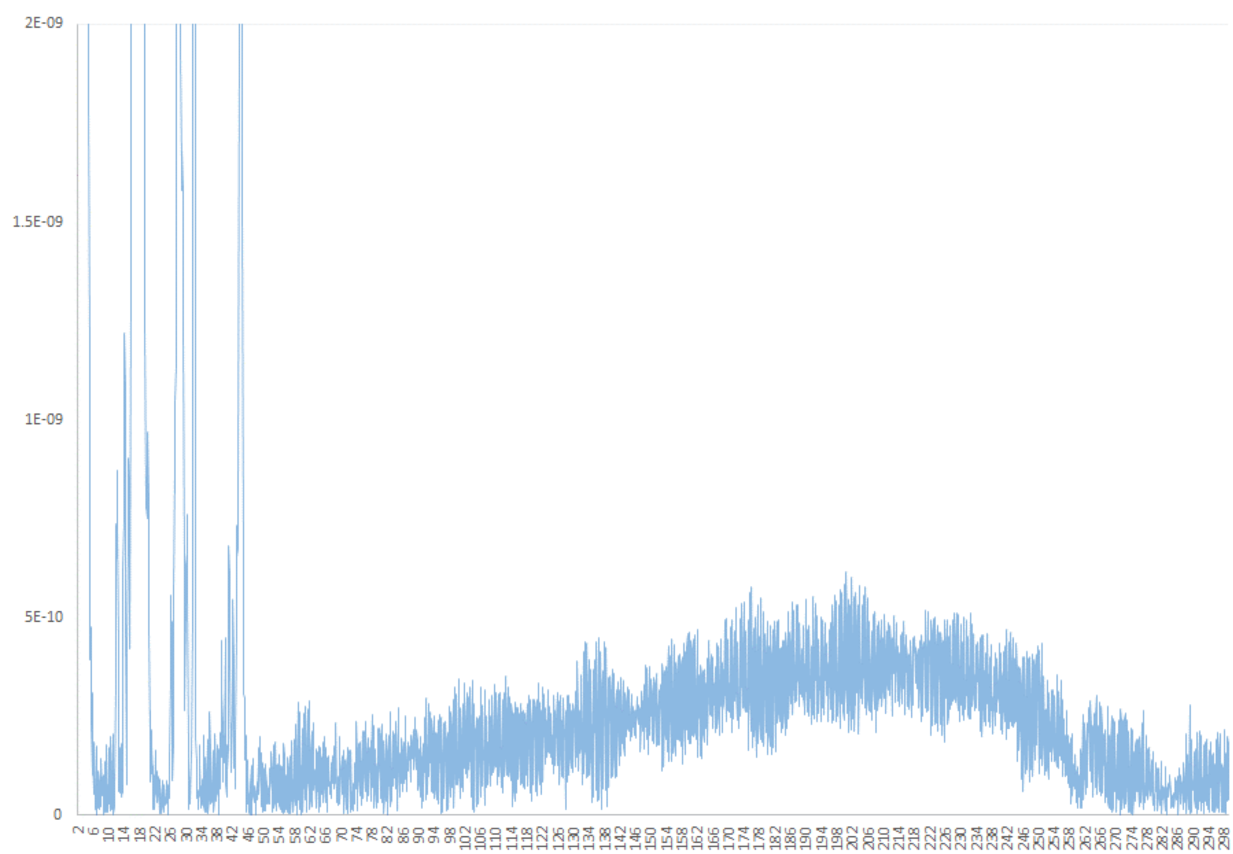



RGA baseline - Helium 100 amu 12-14-20
5E-09
4E-09
3E-09
2E-09
1E-09
0

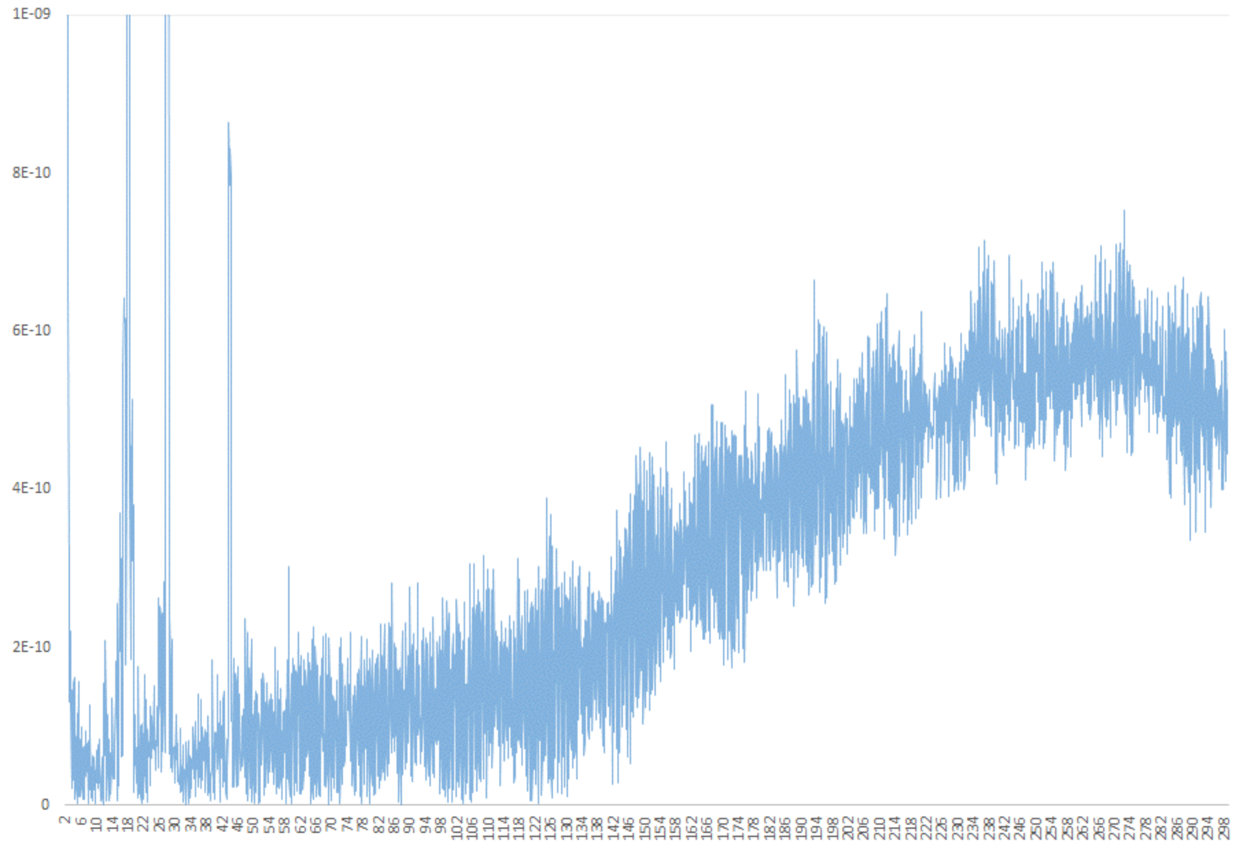
RGA Baseline - Vacuum 12-21-20



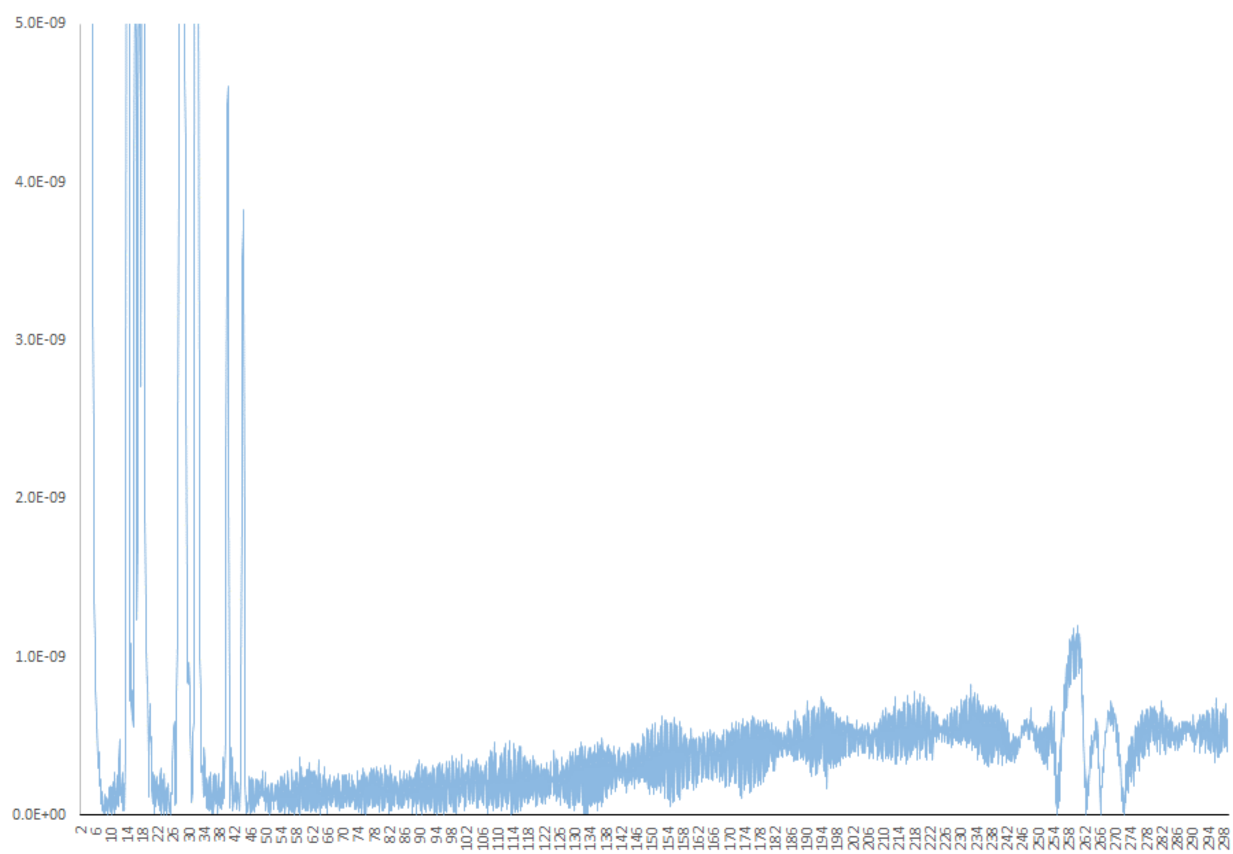
RGA Helium Blower ON 12-21-20

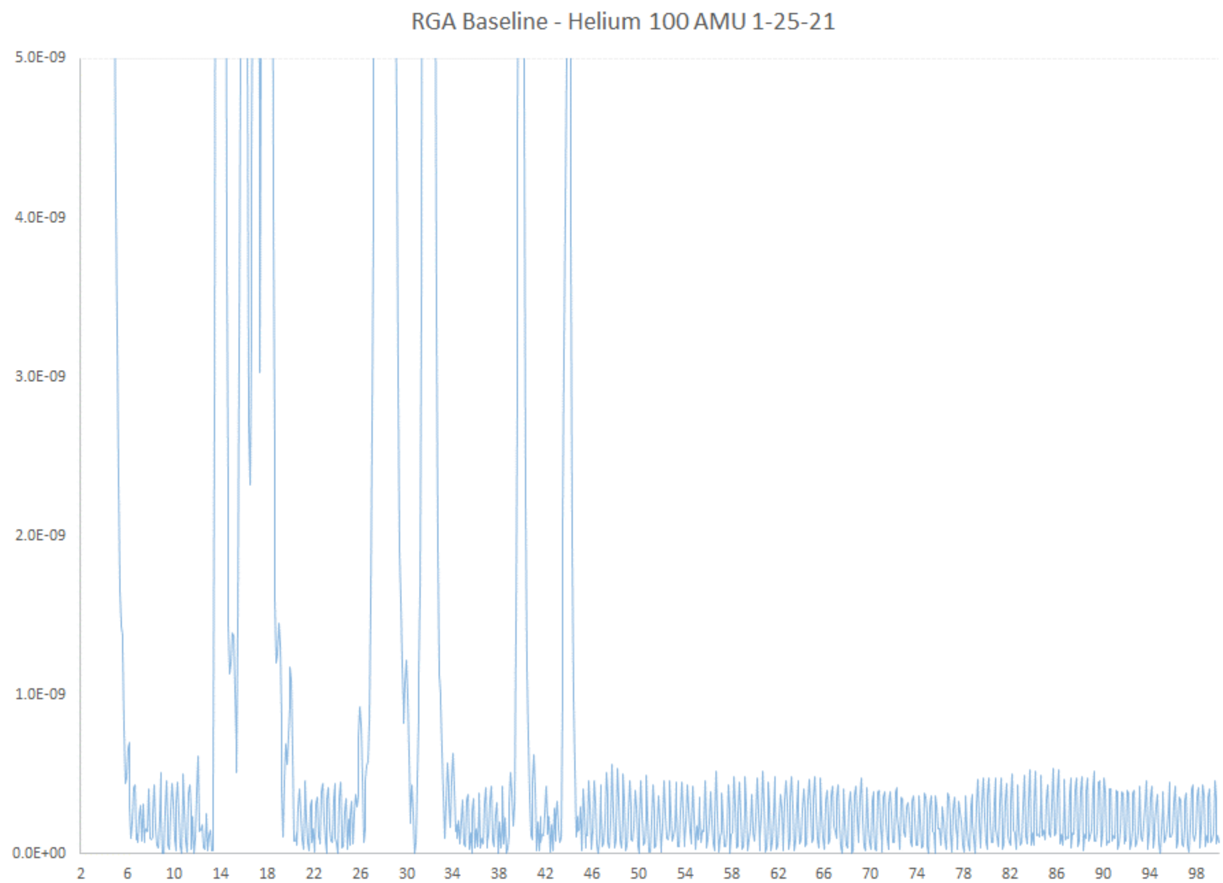


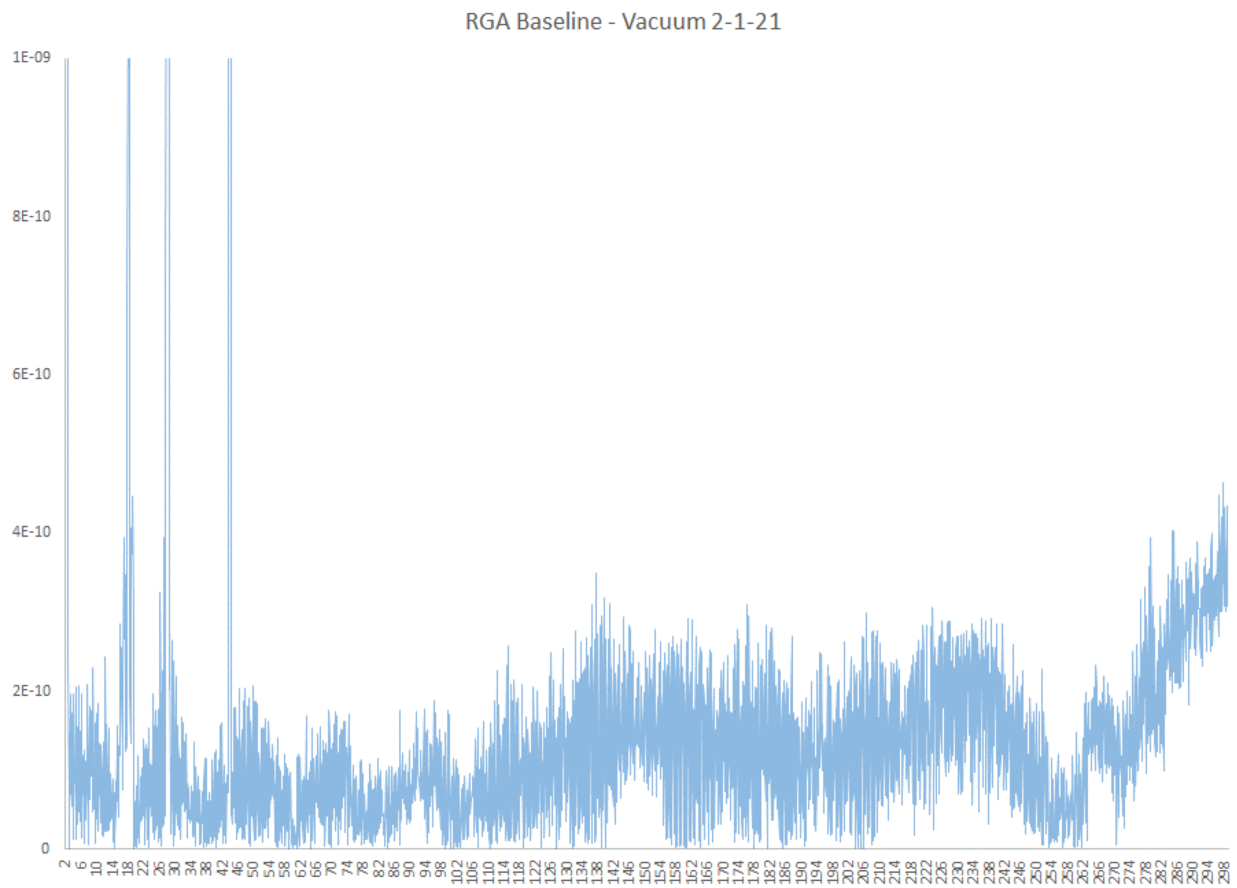
RGA BAseline - Vacuum 1-25-21

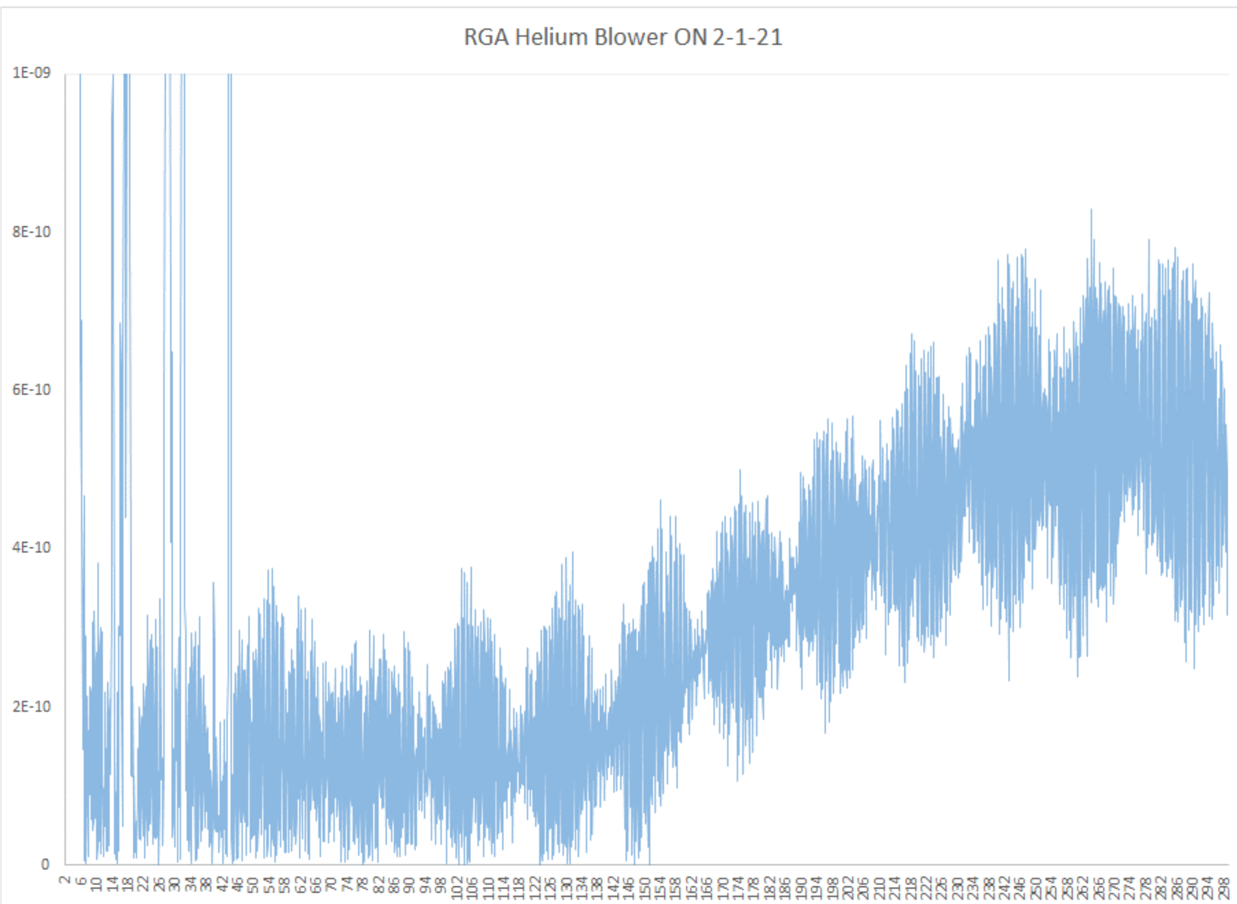


RGA Baseline - Helium-1-25-21

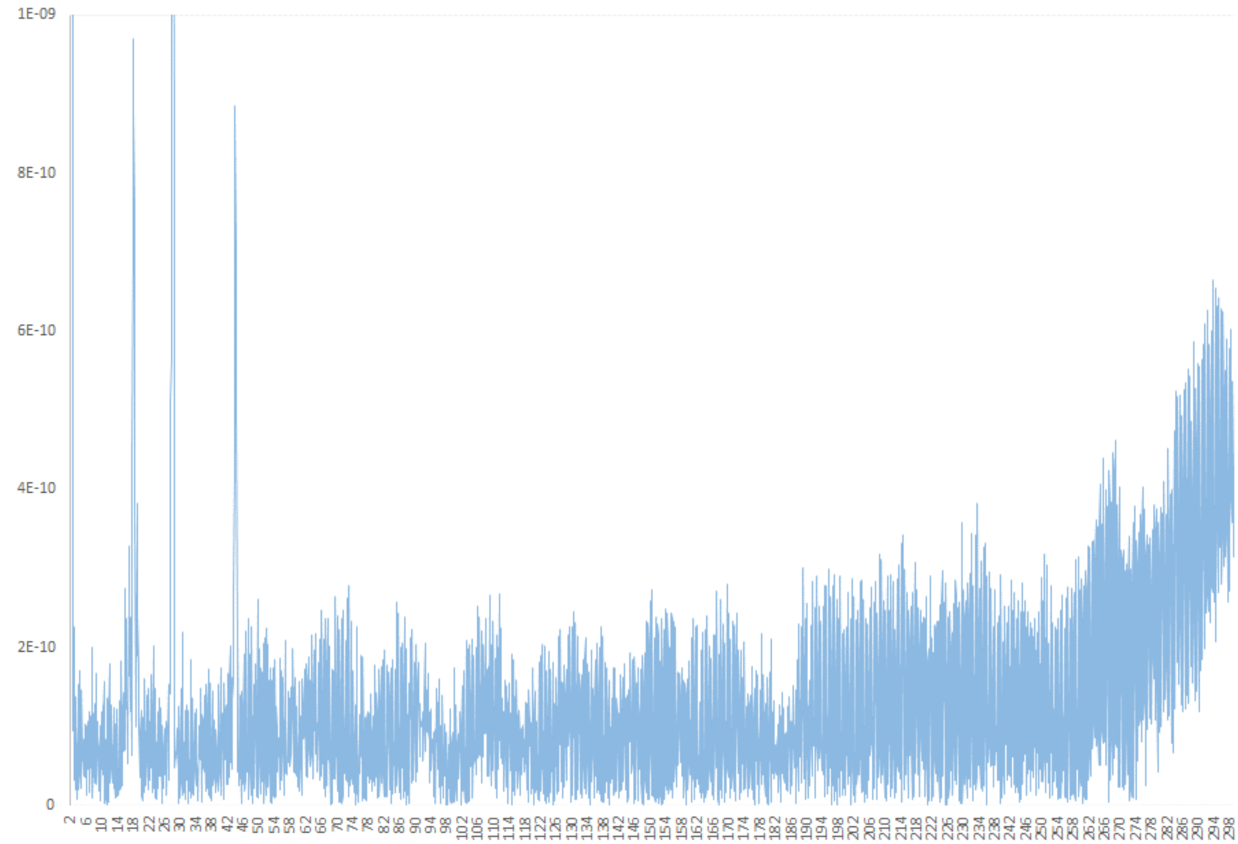



RGA Baseline - Helium 100 AMU 1-25-21
5.0E-09
4.0E-09
3.0E-09
2.0E-09
1.0E-09
0.0E+00
2
6
10
14
18
22
26
30
34
38
42
46
50
54
58
62
66
70
74
78
82
86
90
94
98

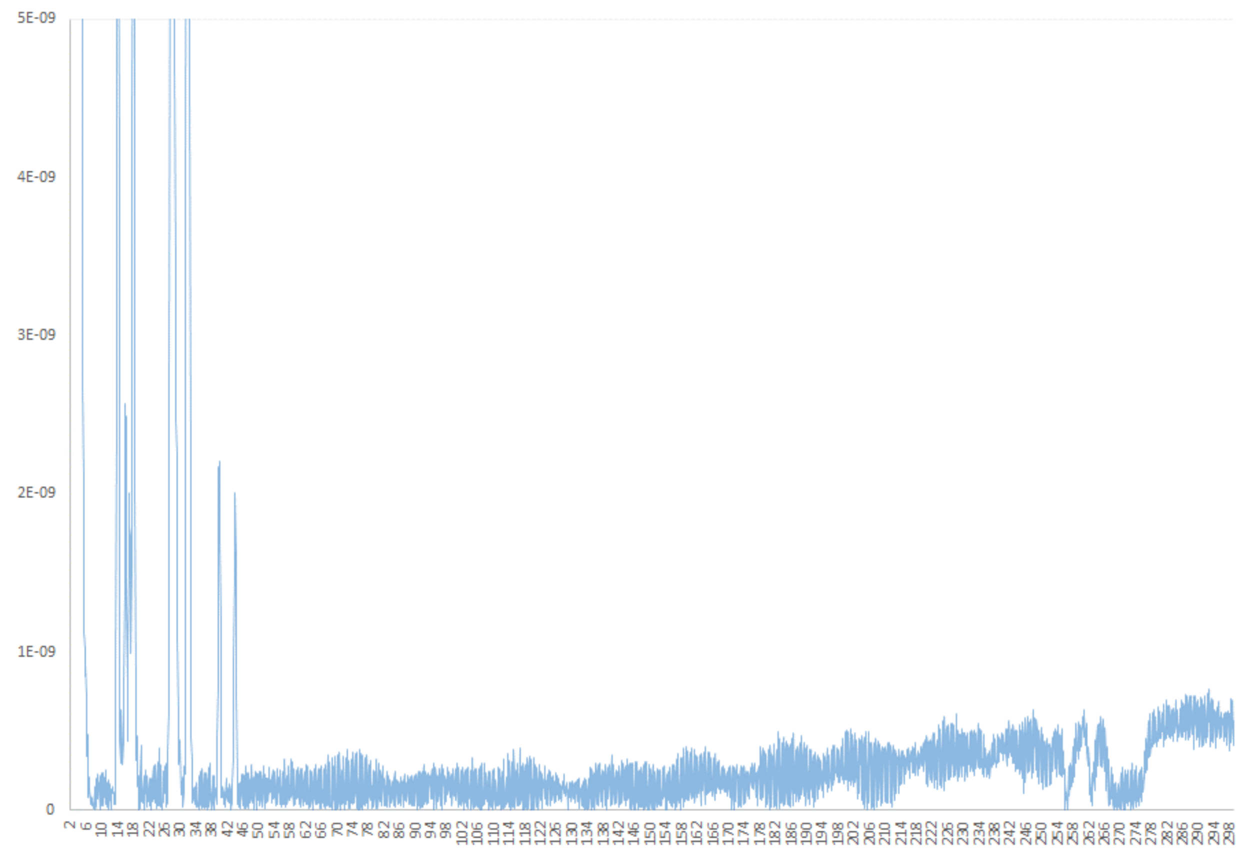

RGA Baseline - Vacuum 2-1-21
1E-09
8E-10
6E-10
4E-10
2E-10
0


RGA Helium Blower ON 2-1-21
1E-09
8E-10
6E-10
4E-10
2E-10
0

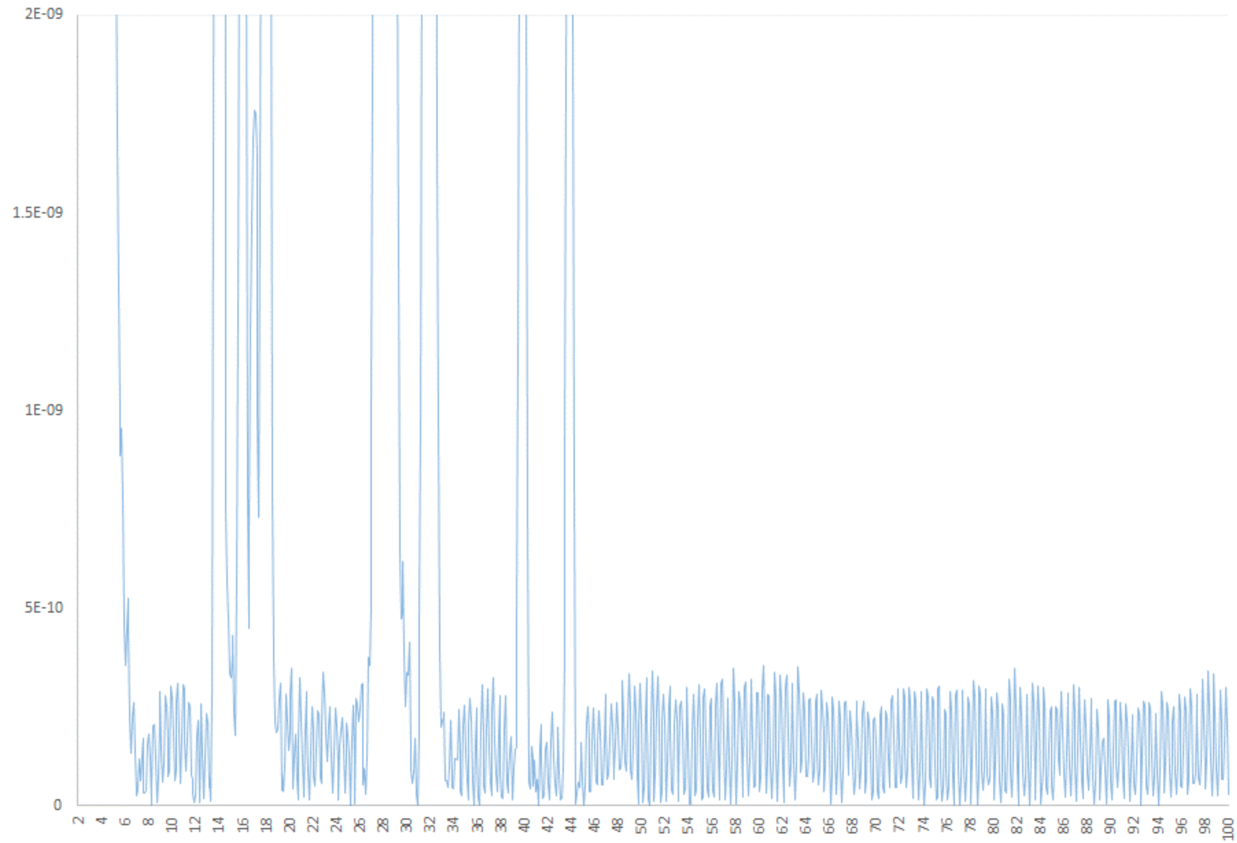
RGA Baseline - Vacuum 2-9-21

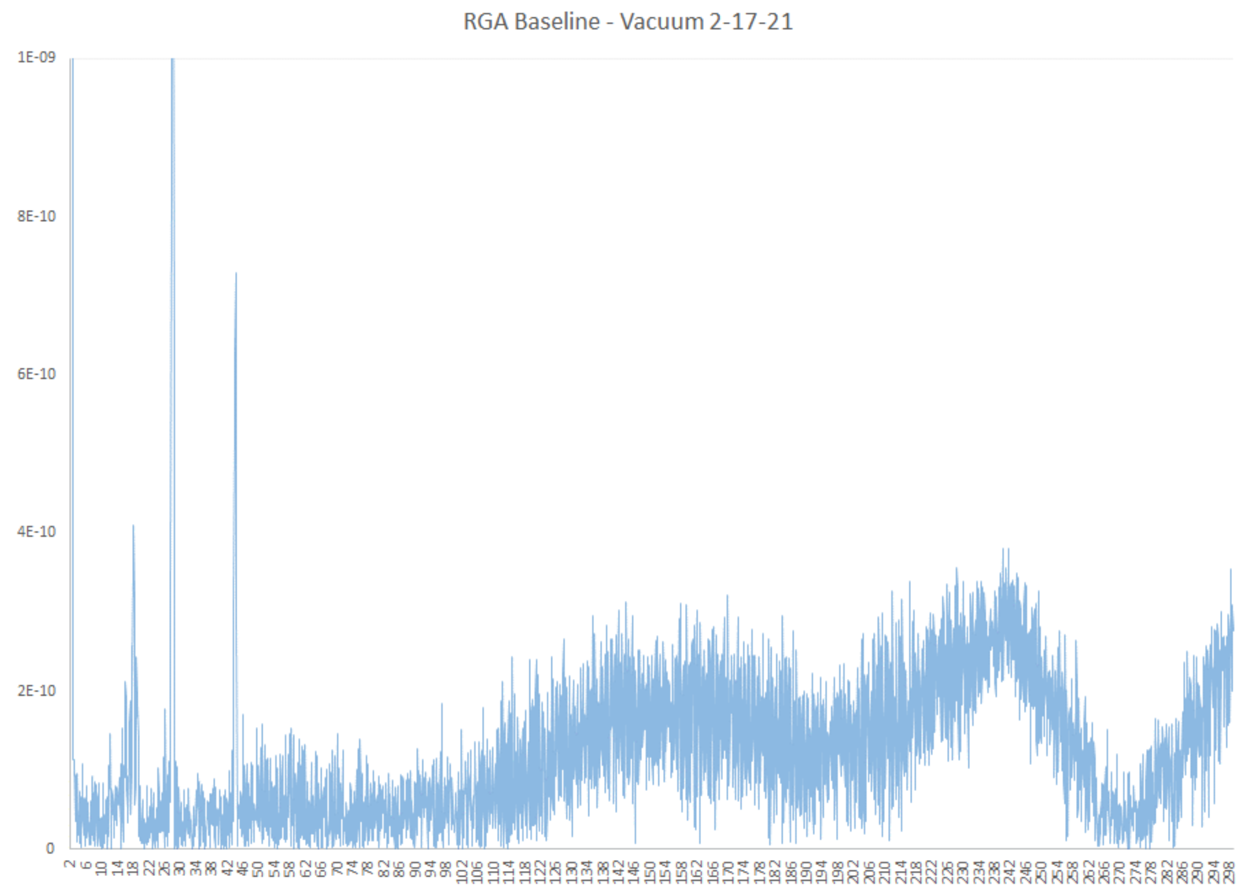


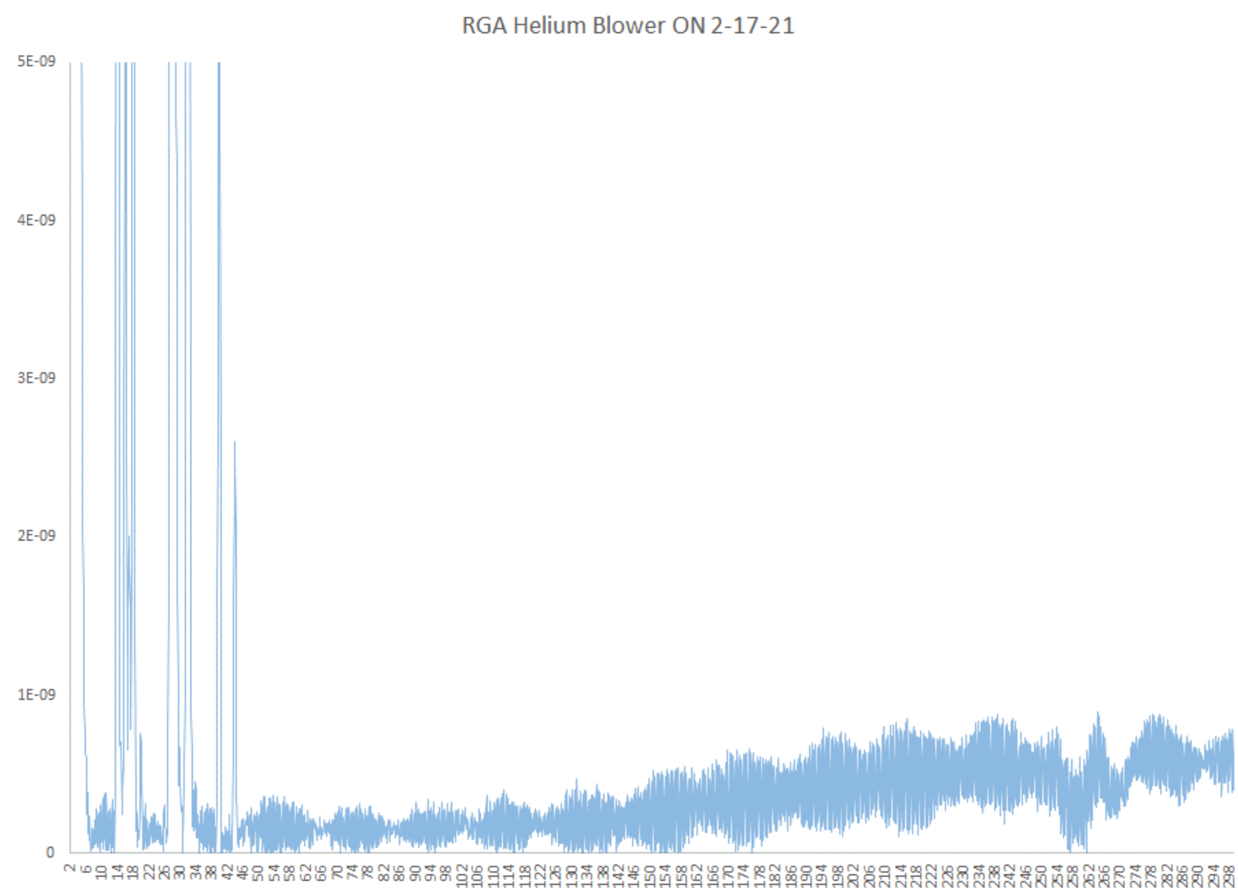
RGA Helium Blower ON 2-9-21

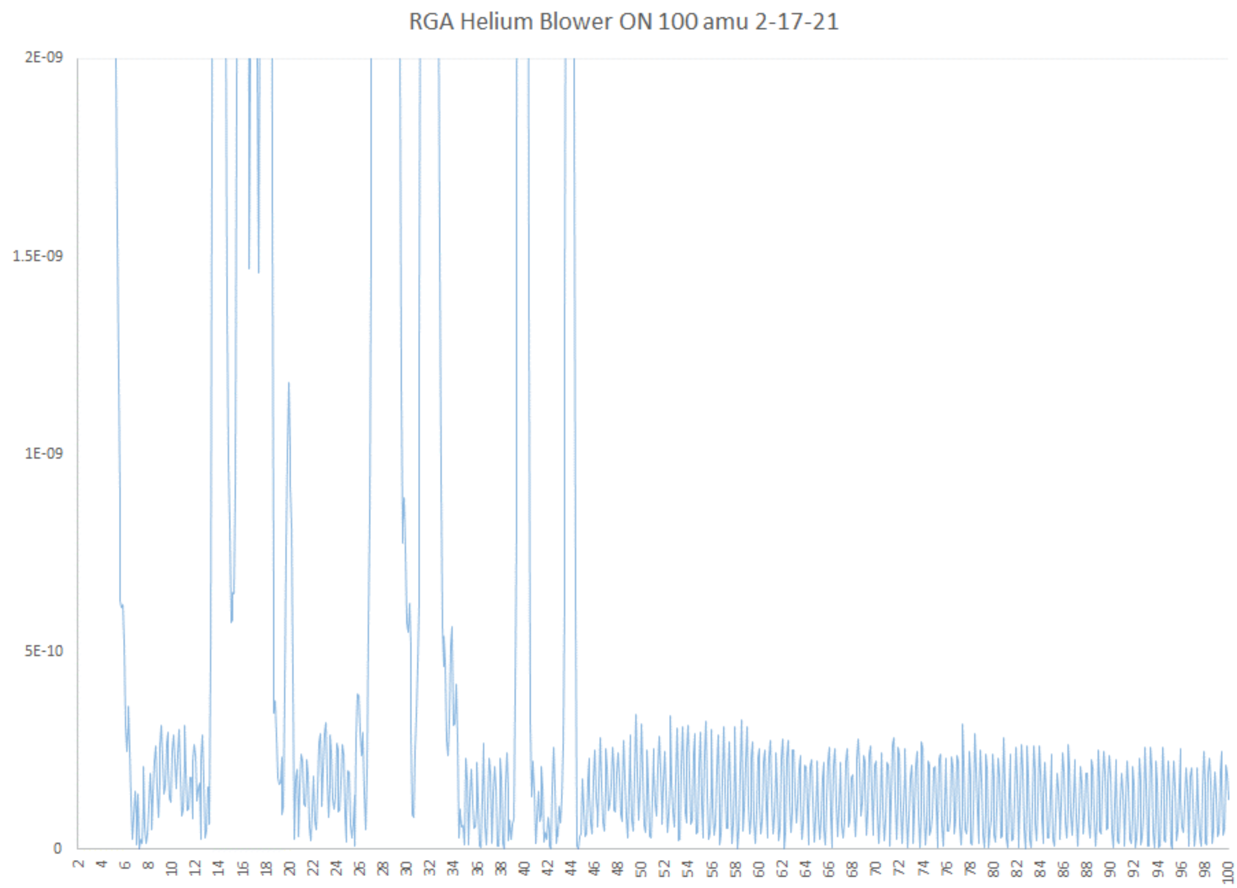


RGA Helium Blower ON 100 amu 2-9-21




RGA Baseline - Vacuum 2-17-21
1E-09
8E-10
6E-10
4E-10
2E-10
0


RGA Helium Blower ON 2-17-21
5E-09
4E-09
3E-09
2E-09
1E-09
0


RGA Helium Blower ON 100 amu 2-17-21
2E-09
1.5E-09
1E-09
5E-10
0
2 4 6 8 10 12 14 16 18 20 22 24 26 28 30 32 34 36 38 40 42 44 46 48 50 52 54 56 58 60 62 64 66 68 70 72 74 76 78 80 82 84 86 88 90 92 94 96 98 100